How to Extract LOW-COST A new process for titanium extraction and production promises to cut costs and expand applications. LOW-COST LOW-COST A new process for titanium extraction and production promises to cut costs and expand applications.

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itanium's high strength, low density, excellent flexibility and strong springback characteristics, high-temperature performance, corrosion resistance, and biocompatibility are highly beneficial in a broad array of structural and specialty applications. However, growth has been severely constrained by today's high-cost, multi-step processes for both primary metal and subsequent parts production. Capital and energy-intensive batch production of primary metal, the need for additional purification steps, multi-step mechanical processing, and high scrap rates are all contributors to high production costs.

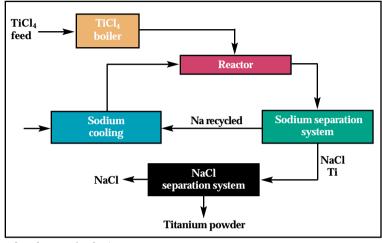
International Titanium Powder (ITP) hopes to eliminate the barriers to growth. The company has developed and is ready to commercialize the Armstrong process, an innovative, low-cost technology for production of high purity titanium metal and alloy powders in a one-step, continuous process. The powder form of ITP titanium enables direct use of highly efficient powder metallurgy processes for manufacture of final product forms. ITP's process, in combination with existing and developing powder processing technologies, will reduce final parts production cost by up to 50%.

This article describes the Armstrong process in detail, and shows how it can economically produce pure titanium metal and alloys for applications such as automobiles and electronics.

The Armstrong process

Most titanium metal today is made by the Kroll process (see box on p. 00), in which titanium tetrachloride is reduced to titanium metal by reaction with molten magnesium. A small amount is also produced with the Hunter process, in which sodium replaces the magnesium. ITP's Armstrong process is based on the same chemistry as Hunter, but there the similarity ends.

Building on their experience with flowing liquid metal in the nuclear industry, ITP researchers designed a process based on a flowing loop of liquid sodium. Controlled continuous injection of TiCl₄ vapor at a single point in the loop allows the reaction to proceed to completion at low temperature, producing small individual particles of titanium metal and co-producing sodium chloride. The particles are removed from the reaction zone by the flowing sodium without allowing growth



Flow diagram for the Armstrong process.

of structures that could trap unreacted raw materials or coproduct and, in turn, reduce purity and performance. The relative flow rates and the geometry of the reaction zone provide control parameters for tailoring the titanium particle shape and size distribution.

At a separate point in the flowing sodium loop, the liquid material is filtered, collecting the solid titanium and salt. When sufficient material accumulates at the filter, the flow is switched to a separate filter without interrupting the sodium flow. The reaction proceeds continuously for both economic efficiency and improved product quality and consistency.

After residual sodium is distilled from the filtrate, the titanium powder is removed and washed to remove the salt. Salt is the only by-product, and it can be broken down electrolytically into sodium and chlorine, and re-used. The washed titanium metal powder directly meets the specifications for commercially pure titanium.

Commercial scale

ITP has operated the Armstrong process in both engineering scale and full commercial scale reactors. The commercial scale pilot reactor now produces quantities for market and applications development, as well as further development of the commercial process design data. A full manufacturing facility will be constructed in the near future.

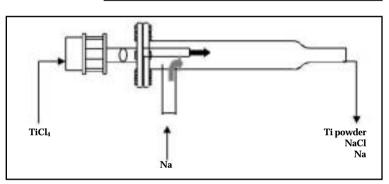
Titanium powder from pilot production runs has been thoroughly tested and analyzed to characterize its quality, morphology, and particle size distribution. ITP is working with powder processors on both process and powder improvements to optimize the use of the company's powder in their processes.

itanium is a silvery white metal stronger than steel but only about half as heavy. It retains its strength at very high temperatures and does not corrode. Titanium is the fourth most common structural metal in the earth's crust. Only iron, aluminum, and magnesium are more abundant. More titanium is available than nickel, copper, chromium, lead, tin, and zinc put together.

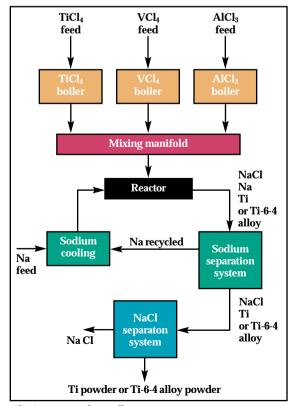
Material properties ^{1,2}

Metal	Weight	Strength	Strength/ Weight ³	Corrosion indices (in sea water)	Life expectancy ¹
Titanium	1.00	1.00	1.00	1.00	Unlimited
Aluminum	0.57	0.29	0.51	0.36	2 years
Steel	1.67	0.59	0.35	0.06	1 year
Stainless Steel	1.67	0.59	0.35	0.31	200 years

1. All material properties are listed relative to titanium, except life expectancy, which lists time for corrosion to penetrate 16-gauge plate exposed to seawater on one side. 2. Treat listed values as representative; properties vary with alloy composition and heat treatment. 3. Strength/weight ratio is an important design parameter. A one pound structural part of titanium is approximately three times stronger than a one pound steel part.



Reactor for the Armstrong process.



The Armstrong direct alloy process.

Direct alloy production

In addition to its cost and product advantages, the Armstrong process technology has another edge over current titanium production methods: Small-scale development work has demonstrated that multiple metal chloride reactants can be fed to the reactor simultaneously to directly produce titanium alloys. The product shows a consistent blend of the

metals in individual particles. Producing a consistent alloy in a single step provides additional freedom in downstream processing. The homogeneity of resulting alloyed end products is difficult or impossible to achieve with conventional melt or blend alloying processes.

ITP's method of alloy production also provides more flexible development of new alloy compositions. Solubility characteristics can limit the range of mixtures that can be achieved in conventional melt processing. This flexibility offers many

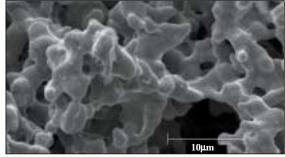
possibilities to develop and supply alloys to meet specific customer needs. In addition to commercially pure titanium, ITP intends initially to commercialize the standard Ti 6-4 alloy (90% titanium, 6% aluminum, 4% vanadium). Other alloys will be added over time for applications requiring a range of properties.

Parts manufacturing

The high extraction cost of titanium metal is but one of the current barriers to broader applications. To make parts, the purified ingots are forged into mill forms such as bars, rods, plate, and sheet. These are machined to final part shapes, an expensive process given the hardness of the metal. The processes also generate significant quantities of scrap, including impurities from tool wear and other handling.

Powder metallurgical processes and new nearnet shape production methods offer a lower cost alternative, with the added advantage of much lower scrap generation. The current downside to this approach is the very high cost of powdered titanium. Powder is currently produced from a melt of purified titanium by a number of atomization processes or by a multi-step hydride-dehydride process.

Unfortunately, the savings made possible via powder-based parts production, are more than



Scanning electron microscope (SEM) image of the titanium product of the Armstrong process.

offset by the high cost of the raw material powder.

Therefore, an extraction process that directly produces very pure titanium powder at costs comparable to that of impure sponge, creates significant new opportunities. The pure starting material eliminates the need for melt purification and the consequent risk of performance problems from contaminant inclusions. Economical application of titanium in a variety of markets ranging from cases for consumer electronics to automotive applications becomes a realistic possibility with affordable titanium powder.

Supply and demand

Today's market for titanium metal is dominated by aerospace applications, creating a boom/bust cycle tied to the fortunes of that industry. The resulting price swings, and especially the high prices when supply is tight, provide an effective barrier to the broader use of titanium. Even at the low end of the cycle, the high cost of parts production via traditional machining of mill forms produced from titanium ingots, inhibits expansion into new markets.

Weak demand has also created difficult times for the titanium production industry. With the closing of the Oremet titanium sponge plant, insufficient operating capacity will likely be available to meet the needs of the next cyclic peak. Yet the high capital costs of the Kroll process cannot be supported in a down market. The next recovery of the aerospace market, coupled with the Department of Defense's intent to build a new generation of titanium-intensive military equipment, will create demand that

cannot be met domestically, and probably not even with available international supply.

Most titanium metal processing to final product shapes generates substantial scrap from remelting, forming, and machining. The overall scrap generation from sponge to final product can exceed ten pounds of scrap per pound of final product in the aerospace industry.

The scrap from the part/form manufacturing steps is collected, cleaned, and recycled to the sponge and ingot alloying process. The supply and pricing of scrap is therefore a major factor in total availability and pricing. The aerospace industry is working hard on technologies to reduce their huge "buy to fly" ratio. With less scrap to recycle, the price of titanium ingot would thereby increase.

Without new supplies of the metal, the same would happen if a large market such as automobiles included even one or two titanium parts in new vehicles. This would cause a large increase in the demand, without increasing the amount of scrap recycle, thus forcing further price pressure.

The Kroll process

The basic chemical process for titanium production was patented by Wilhelm Kroll in 1938. The Kroll process has three stages: primary reaction, sponge handling, and melt purification. The primary reaction is carried out in a large retort where titanium tetrachloride is sprayed onto a high-temperature reactive metal surface. It reacts to form a sintered, porous mass of titanium, salt, and unreacted chemicals called "sponge."

After the reaction reaches its equilibrium state (over a few days duration), the retort is sparged with a high temperature helium sweep, then cooled and finally passivated by blowing gas through the retort. One end of the retort is cut off and the sponge is jacked out of the retort. It is then chopped and ground into chips of about one centimeter size. The chips are acid-leached, water-washed, and dried.

In the final stage, the chips are compressed and welded into an electrode, which in turn is melted into an ingot in a vacuum arc furnace. Alloys of titanium are formed by adding chips of the alloying elements to the electrode as it is being compressed and welded into an electrode shape. The arc melting removes volatile impurities and improves homogeneity. **High purity alloys require an initial melt and two further remelts.**

The Kroll process has a number of serious disadvantages:

- It is a multi-step process.
- Each individual step is a batch process.
- The initial stage operates at high temperature in retorts with short working lifetimes.
 - It produces a sintered product with high levels of impurities.
- The second stage removes some of the impurities and discharges a polluted waste stream.
- The third stage removes the rest of the impurities through multiple melts in high vacuum arc furnaces.

All of these inherent disadvantages add up to a product that is so expensive it can compete with other metals only in very specialized niche markets.

Two methods are used to produce titanium powder from titanium ingots: hydride/dehydride and molten metal fragmentation. In the hydride/dehydride process, the ingot is exposed to hydrogen under conditions in which it forms a brittle titanium hydride. The brittle hydride is crushed to the required powder size, and then the conditions are altered to remove the hydrogen, thus producing titanium powder.

In the molten metal fragmentation process, the titanium is melted and fragmented, either by centrifugal force on the rim of a spinning titanium disk, or by gas jet disruption of a molten titanium stream.

As with the process to produce titanium ingot, the processes required for titanium powder are also complex and inherently expensive. This forces the prices to be rather high and results in a very small market for titanium powder.

Consensus of industry leaders is that any new investment in the current process would be a financial challenge because of the risky cyclical market and the high capital costs of the Kroll process. A new extraction process is needed with lower capital and operating costs.

Only ITP's Armstrong process has been demonstrated at full scale in a pilot plant and is close enough to commercialization to be timely in meeting growing demand. Its low capital cost and efficiencies will break the titanium boom/bust cycle and provide consistent affordable titanium and lower cost parts production for the emerging markets.

International Titanium Powder has Armstrong powder available for evaluation and application development by qualified parties.

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